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ABSTRACT

The project goal is the development of novel damage assessment devices for carbon fiber-reinforced composite structures utilizing carbon-fibers as self-sensing sensors. Two key components of this project are the fabrications of electrodes required for electrical inputs/outputs through the composites, and the development of data-processing scheme to diagnose the damage state. The former takes the advantage of new technology based on the thermal spray to directly deposit onto composite laminate surfaces to fabricate network of electrodes. Several conditions were tested and optimal process was identified. The latter task utilized simulations to develop the post-processing approach and preliminary results were promising.

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'Self-Sensing Fibers and Thermal Sprayed Electrodes for Health Monitoring of Composites', 49th Annual Technical Meeting of Society of Engineering Science Georgia Tech, Atlanta, October 2012.

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Assessment of Impact Damage in Composites via Self-Sensing Fibers

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Department of Mechanical Engineering
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Abstract

The project goal is the development of novel damage assessment devices for carbon fiber-reinforced composite structures utilizing carbon-fibers as self-sensing sensors. Two key components of this project are the fabrications of electrodes required for electrical inputs/outputs through the composites, and the development of data-processing scheme to diagnose the damage state. The former takes the advantage of new technology based on the thermal spray to directly deposit onto composite laminate surfaces to fabricate network of electrodes. Several conditions were tested and optimal process was identified. The latter task utilized simulations to develop the post-processing approach and preliminary results were promising.

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- Fig. 6. Masks with different patterns were used to make electrodes (spraying through masks).
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- Fig. 10. Resistances between the central electrode G and the neighboring electrodes. As measured and adjusted with estimated contact resistances are shown.
- Fig. 11. Changes in resistances due to damage (12mm diameter hole) between electrode pairs are illustrated with arrow thickness. For clarity, the measurements from 4 electrode and 9 electrode grids are shown separately.
- Fig. 12. Square composite model with 4 electrodes on the front surface containing damage on the back surface. The coordinate and the damage parameters are noted.

Statement of Problem

The present project was focused on developing an innovative health monitoring system based on self-sensing fibers of composites, whose inherent heterogeneous microstructures make detections of potential damage difficult. The proposed system utilizes recently developed thermal spray technology to synthesize electrodes. Currently available technologies require complex processes and are not suitable to fabricate numerous electrodes necessary for large-scale sensor systems.

In our efforts, two issues critical to fabricating reliable TS electrodes were closely studied. They are the good electrical conductance as well as the good adhesion strength between TS deposited electrodes and composite laminates. In order to achieve optimal fabrication process to establish reliable electrodes, electrodes were deposited under various TS conditions and also different surface pre-treatment. For each condition, the electrodes were tested for

- □ Electrical resistance with neighboring electrodes
- □ Adhesion strength with composite laminates.

The lower "contact resistance" at the interface of electrodes and carbon fibers is needed to process measured data and reduce variability among different electrodes. The second is needed to fabricate structurally sound electrodes during service. These two conditions are influenced by the thermal spray process as well as pre-surface treatment.

Robust damage monitoring system also requires accurate data interpretation schemes. As resistance changes are measured at various pairs of electrodes, they must be processed and the state of potential damage must be estimated. The current project uses numerical analysis to generate simulated conditions and use the resulting resistances to re-construct damage state. Since the actual damage state is known, the accuracy of estimated state can be evaluated. The procedure is consisted of;

- □ Generate simulated resistance measurements from finite element model with damage.
- □ Process the indirect measurements via inverse analyses to estimate damage.

The simulation study to predict and tailor the damage monitoring system is critical in this project. Without references to estimated resistances, it would be impossible to interpret the measurements in actual samples. The computational results are also necessary in developing a suitable data processing scheme since the resistance change due to damage can be small.

Summary of Results

Thermal Spray Electrode Fabrication Processes

In the projects, electrodes are fabricated onto composite laminate surface using the following three thermal spray techniques.

- 1. **Twin-wire arc** spray with zinc and copper layers.
- 2. High Velocity Oxygen Fuel (HVOF) with copper powder.
- 3. Atmospheric **Plasma Spray** with copper powder.

The first process uses a pair of electrically conductive wires (<u>www.vividinc.com/newSite/tsp-wirearc.shtml</u>) and produces denser coatings at lower temperature (~1,000°C) than the plasma

spray. The second process (HVOF) relies on high flow of oxygen to generate high temperature and particle velocity

(www.gordonengland.co.uk/hvof.htm

). The last process, the plasma spray, produces the highest temperature (~3,000°C) with ionization via high voltage discharge between anode and cathode

(www.vividinc.com/newSite/tsp-

plasma.shtml). All processes were conducted at the Center for Thermal Spray Research (www.ctsrsunysb.org) Stony Brook at University. To ensure a good adhesion of coatings to laminate with the lower temperature, zinc was initially sprayed and then copper was deposited in the twin-wire arc process. Many previous tests have shown excellent bonding strength of zinc to any surfaces. The HVOF process was expected to produce dense Cu coatings with good adhesion. However, several test revealed the particle velocity is too high for composite laminate and damage the substrate. Particles also bounced off from the surface. The process will be further tuned in the future study to test its capability. As HVOF, with the plasma spray (APS) process, copper was directly sprayed onto the laminate using programmed robot as shown in



Fig. 1. Molten copper is plasma sprayed directly onto composite laminate over a mask to fabricate electrodes.

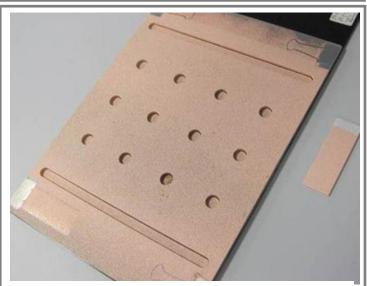


Fig. 2. Copper particles are plasma sprayed over an aluminum mask which covers composite laminate and electrodes are directly fabricated through open holes.

Fig. 1. It was expected that the high temperature of plasma removes or evaporate epoxy phase and generates good adhesion with carbon fibers. In all processes, copper was sprayed over a mask with holes for electrodes as shown in Fig. 2.

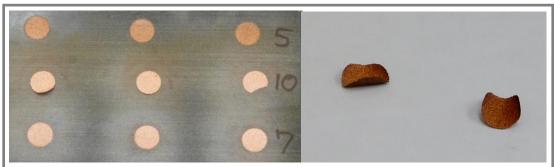


Fig. 3. Adhesion of electrodes on composite is tested with various spray passes. Some Cu electrodes are self-delaminated due large thermal stress as shown.

Two types of composite panels were prepared and cut to 12" by 9" dimensions. Both types were 8-ply with $[0^{\circ}]_{8}$ and $[0^{\circ}/90^{\circ}]_{2s}$ arrangements with ~2mm thickness. In all specimens, their surfaces were initially grit-blasted to remove relatively thick (~50 μ m) epoxy only layer.

Adhesion of electrodes to the substrate laminate is a major problem in TS electrodes. In order to determine an optimal process to obtain good bonding strength, several tests with various spray parameters; powder feed rate, particle temperature and velocity, stand-off distance, raster speed and number of spray passes. For an example, as shown in Fig. 3, many spray passes (10) caused large thermal stresses to develop during cool down and cause partial delamination while a few spray passes do not deposit sufficient thickness. Also a high particle temperature and a large rate of power feed resulted in complete delamination as shown. After several trials, optimized parameters were found and electrodes were fabricated as shown in Fig. 4.

The first process uses a pair of electrically conductive wires (www.vividinc.com/newSite/tsp-wirearc.shtml) and produces denser coatings at lower temperature (~1,000°C) than the plasma spray. The second process (HVOF) relies on high flow of oxygen to generate high temperature and particle velocity (www.gordonengland.co.uk/hvof.htm). The last process, the plasma spray, produces the highest temperature (~3,000°C) with ionization via high voltage discharge between anode and cathode (www.vividinc.com/newSite/tsp-plasma.shtml). All processes were conducted at the Center for Thermal Spray Research (www.ctsr-sunysb.org) at Stony Brook University. To ensure a good adhesion of coatings to laminate with the lower temperature, zinc was initially sprayed and then copper was deposited in the twin-wire arc process. Many previous tests have shown excellent bonding strength of zinc to any surfaces. The HVOF process was expected to produce dense Cu coatings with good adhesion. However, several test revealed the particle velocity is too high for composite laminate and damage the substrate. Particles also bounced off from the surface. The process will be further tuned in the future study to test its capability.

Various Surface Pre-Treatments

Prior to TS electrode depositions, surfaces of composite laminated were pretreated under conditions. Optical micrographs of some of them are shown in Fig. 4.

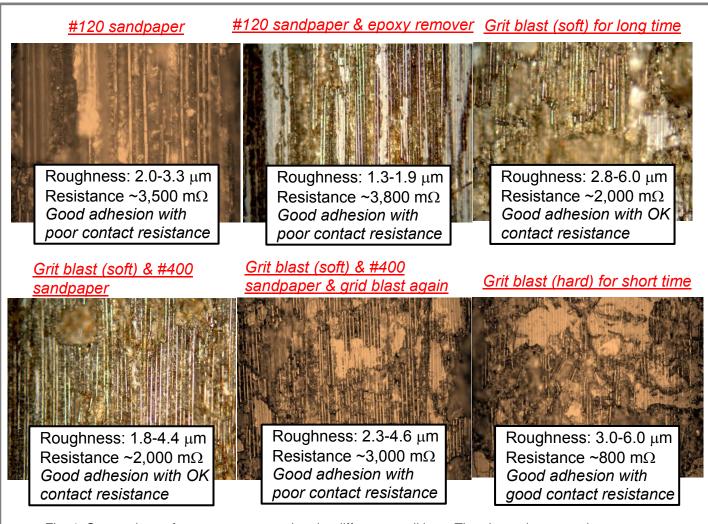


Fig. 4. Composite surface were pretreated under different conditions. The electrodes were plasma sprayed. After wiring was soldered, resistances with neighboring electrodes were measured.

In some, liquid epoxy remover was used to clean away residual epoxy fragments. However in most cases, the resulting contact resistances were worsened after the chemical liquid was used. It appears that the remover may form thin insulating layer on the carbon fibers upon its applications. Aside from grit blasting, the surface was also pretreated with sandpapers with various grits. These abrasives are used to remove the epoxy layer to essentially exposed carbon fibers. For grit blasting with alumina particles, the pressure level was also varied. At hard/high pressure, the particles not only removed the epoxy but also broke the carbon fibers as shown in Fig. 4. We have also performed combination of sandpaper and grit blasting to see the combined effects.

After the surfaces were pretreated, electrodes were fabricated using plasma spray with mask as discussed in the previous report. Figure 5 shows some of deposited electrodes. If the bonding strength is low, some of electrodes delaminated due to residual stress developed by the thermal coefficient mismatch as shown. For un-delaminated electrodes, two wires were soldered to

perform 4-point-probe with a low current meter (Keithley, Inc.) which offsets instrumental contact resistances. These results are described detailed in Appendix.



Fig. 5. Some of plasma deposited electrodes. If the bonding strength is low, they were self-delaminated during cooling. They were wired to measured resistances.

Optimal Electrode Fabrication Process

After testing many differently prepared electrodes, one of optimal surface pre-treatment is that grid blasting with weak pressure for a few second (until the color on the surface is changed) and then rubbing 7 times along the fiber direction with sand paper #240. This process gives good adhesion strength as well as sufficient contact condition with carbon fibers. Alternately, short time grit blasting under high pressure also produces good strength as well as contact. At first glance, this appears to break so many carbon fibers, but the overall contact condition is good as shown by lower measured resistances between adjacent electrodes.

Testing with Artificially General Damage

As carried out earlier, a hole representing damage was drilled in some of composite plates with TS electrodes. For testing, three different composite laminates were acquired. Two crossply laminates with $[0^{\circ}/90^{\circ}]_{2s}$ and $[0^{\circ}/90^{\circ}]_{4s}$. The thickness of the former type of plate is 1.6mm while the latter is 3.2mm. Also a laminate with unidirectional fiber $[0^{\circ}]_{4s}$ with 1.6mm plate thickness was also tested. We have also utilized different mask design as shown in Fig. 6 to measure remittances among different electrodes.

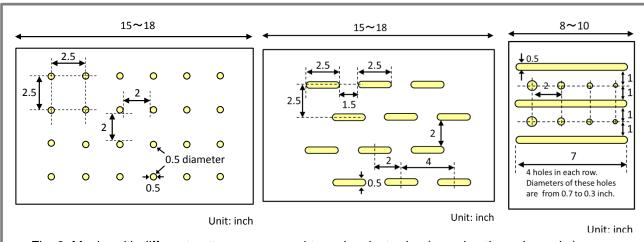


Fig. 6. Masks with different patterns were used to make electrodes (spraying through masks).

In each composite laminate, a 10 mm diameter hole was drilled through-thickness to resemble damage as shown in Fig. 7. The size and depth of the hole was chosen arbitrary but made large enough to detect resistance changes due to damage. Since relatively large errors and noises are expected in this preliminary test, the measurements need to be beyond estimated errors. Thus a through-thickness hole, unlike part-through damage as analyzed in the simulation study was selected. In subsequent study, various types of damage will be tested to assess the determination capability of the proposed system. The results are summarized in our published paper [1].

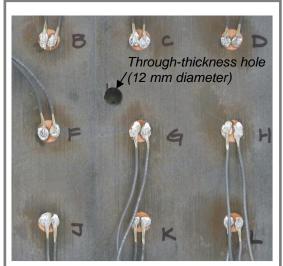


Fig. 7. A 0.5" (12 mm) diameter hole was drilled to measure its influence on electrical resistances among electrodes.

Measurements - Contact Resistances

Resistances between electrodes (Fig. 8) were measured using a low current meter (Keithley, Inc.) with four-point probe method, which offsets instrumental contact resistances. Each electrode is soldered with two wires as shown in Fig. 7. First, resistances between each electrode and base electrodes at the top and bottom (2 locations each) are measured to determine their variations. Since the effective distances from 4 wiring locations on the base electrodes are approximately same for each electrode, the averaged values should offer some information on the contact resistances between electrode and carbon fibers. It is expected the resistances between two electrodes to be comprised of the carbon fiber resistance and the interface resistance between carbon fibers and copper electrodes. Although the latter effects can be subtracted when the difference in resistances due to damage are computed, large contact resistances can increase

measurement error or noise. Raw measurements of resistances among four separate panels are shown in Fig. 9. For the twin-wire arc electrodes, variations of more than $100m\Omega$ are observed in both unidirectional and cross-ply panels. The resistance variations are about half as much in the plasma spray electrodes. addition, magnitudes In the resistances of twin-wire arc were generally much higher than those of plasma spray. These results imply better contact conditions with the electrodes fabricated with plasma spray than those made with twin-wire arc. Much higher deposition temperature contributed the better bonding condition.

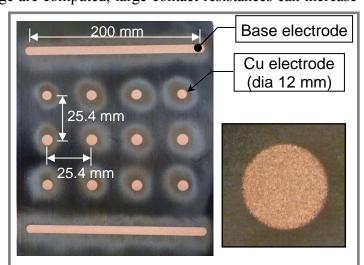


Fig. 8. Grid of electrodes on composite laminate fabricated by plasma spray. Magnified view of an electrode is also shown.

In order to test the consistency of fiber resistances, resistances between electrode grids were measured next. Here the central electrode was chosen and resistances between the central electrode and the neighboring electrodes were measured. Figure 10 shows the results obtained from the plasma spray electrodes on the cross-ply $[0^{\circ}/90^{\circ}]_{2s}$ panel. Here the central electrode is chosen as the one labeled G and pink bars correspond to "as measured" results. These values were adjusted with contact resistances estimated from the measurements shown in Fig. 9, and shown as green bars in Fig. 10. With the adjustment, more consistent results (e.g., similar resistances for GB, GJ, GD and GL pairs) were obtained. Although not shown here, the similar analyses of the other three panels were also conducted. In the unidirectional $[0^{\circ}]_{8}$ panel2, there should be two low resistances and six approximately similar resistances. The as-measured data indeed showed such behavior with some inconsistency. The adjusted or calibrated results exhibited better agreements with the predicted results based on the fiber direction. A similar measurements are also made with the twin-wire arc cross-ply $[0^{\circ}/90^{\circ}]_{4s}$ panel. These results are

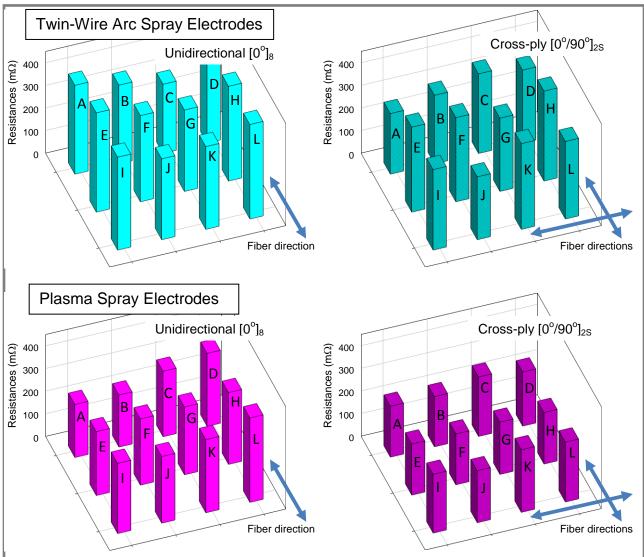


Fig. 9. Raw resistance measurements of four separate laminate, unidirectional and cross-ply panels with for twinwire arc and plasma spray made electrodes. Each value represents the average resistance between an electrode to the base electrodes.

consistent with the ply-arrangement of these panels.

<u>Measurements – Resistance Change due to</u> Damage

In an initial step toward monitoring damage in actual composite laminate, a 10 mm diameter hole was drilled throughthickness to resemble damage. The size and depth of the hole was chosen arbitrary but made large enough to detect resistance changes due to damage. Since relatively large errors and noises are expected in this preliminary test, the measurements need to be beyond estimated errors. Thus a through-thickness hole, unlike part-through damage as analyzed in the simulation study was selected. In subsequent study, various

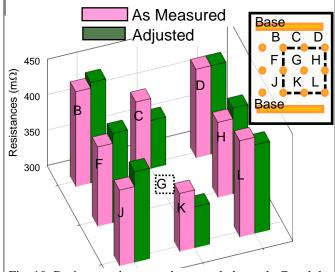


Fig. 10. Resistances between the central electrode G and the neighboring electrodes. As measured and adjusted with estimated contact resistances are shown.

types of damage will be tested to assess the determination capability of the proposed system. Note that the post-process to estimate the damage was not carried out in this preliminary experiment since it would require additional information on the resistivity of each ply within the laminate which was available yet. Furthermore the data process also requires many computations to set up the reference/forward solutions. The aim is to simply verify the changes in resistances due to an existence of a hole in this preliminary phase.

In this experiment, plasma spray electrodes on the cross-ply $[0^{\circ}/90^{\circ}]_{2s}$ panel was chosen. Prior to drilling, eleven electrode pairs in 3 by 3 electrode grid were selected and their resistances were measured (without damage). Their values range between $401\sim500$ m Ω . Then

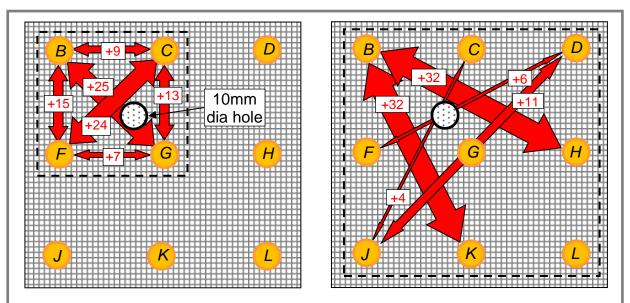


Fig. 11. Changes in resistances due to damage (12mm diameter hole) between electrode pairs are illustrated with arrow thickness. For clarity, the measurements from 4 electrode and 9 electrode grids are shown separately.

the hole was drilled, and the resistance measurements were again made. Since the electrical flow is obstructed with the hole, the resistances between electrode increased by $4\sim32~\text{m}\Omega$. Note we estimate the measurement error bound to be $5\sim8~\text{m}\Omega$ in these test. The changes in resistances are graphically shown in Fig. 11. As expected, large resistances were observed in electrode pairs whose paths cross or run near the drilled hole (BG, CF, BH, BK pairs). However some inconsistencies were also observed. For examples, the CJ and DF pairs showed only marginal increases even though their paths pass through the hole.

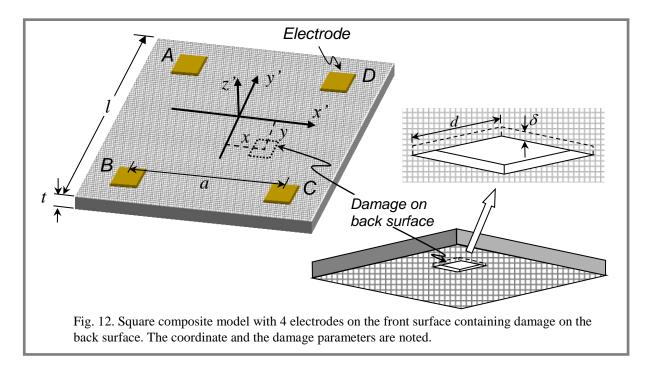
During the next phase, additional investigations will be made on the various thermal spray processes. Although statistical nature of resistances measurements can be accommodated in the data analysis, lower and more consistent contact resistance (between fibers and electrodes) will be ideal. Furthermore, an improve procedure to measure resistances to lower the error or noise will be developed.

Data Interpretation Scheme

Development on robust data processing was also carried out during the period. Here the inverse analysis to measured resistances was refined and the 3D finite element model to simulate and verify the procedure was also carried out. Damage was modeled as shown in Fig. 10 and 3D FE mesh containing 160,000 8-node elements was constructed. Here four damage parameters, d, δ , x and y, are used to define the state of damage. In the inverse analysis, reference solutions were constructed using the cubic Lagrangian interpolation functions. Suppose the electrical residence value at α th electrode pair, $R_{\alpha}(x, y, d, \delta)$, is expressed as a continuous function of damage parameters as,

$$R_{\alpha}(x, y, d, \delta) \approx \sum_{i=1}^{4} \sum_{j=1}^{4} \sum_{p=1}^{4} \sum_{q=1}^{4} R_{\alpha}(x_i, y_j, d_p, \delta_q) N_{ijpq}(x, y, d, \delta),$$
 (1)

where the interpolation function is defined as,



$$N_{ijpq}(x, y, d, \delta) = \prod_{m=1, m \neq i}^{4} \frac{x - x_m}{x_i - x_m} \prod_{n=1, n \neq j}^{4} \frac{y - y_n}{y_i - y_n} \prod_{l=1, l \neq p}^{4} \frac{d - d_l}{d_p - d_l} \prod_{r=1, r \neq q}^{4} \frac{\delta - \delta_r}{\delta_q - \delta_r}.$$
 (2)

In the above, x_i and y_i are *i*th and *j*th sample point within the range of damage location, and d_p and δ_q is *p*th and *q*th sample point within the range of damage size, respectively. N_{ijpq} denotes the cubic Lagrangian interpolation function. To extract the most probable damage parameters, the difference between the measured electrical resistance and the estimated one calculated from estimated damage parameters is minimized. In this study, the error object function for *n* electrical potential measurements is introduced as,

$$\varphi(x, y, d, \delta) = \frac{1}{n} \sum_{\alpha=1}^{n} \left(\frac{R_{\alpha}(x^{\text{est}}, y^{\text{est}}, d^{\text{est}}, \delta^{\text{est}}) - R_{\alpha}^{\text{meas}}}{R_{\alpha}^{\text{ref}}} \right).$$
(3)

Here, x^{est} and y^{est} are estimated damage location in the x' and y' coordinates, and d^{est} and δ^{est} mean estimated damage size and depth, respectively. To find the minimum value of the error objective function, the downhill simplex method is used. This method is known as a one of the most popular methods for multi-dimensional optimization especially in case of derivatives of objective function are either unavailable or discontinuous. It was introduced by Nelder and Mead [2] and previously utilized to find the embedded delamination in composite [3]. The method is based on the concept of a simplex, which is a polytope of N+1 vertices in N dimensions. Since there are four unknown parameters in the present work, the value of N is four. 4. The detail can be found in [3].

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- [1] M. Ogawa, C. Huang and T. Nakamura, Damage detection of CFRP laminates via self-sensing fibers and thermal sprayed electrodes (2012), *Nondestructive Testing and Evaluation*. DOI:10.1080/10589759.2012.665919.
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- [3] N. Ramanujam, T. Nakamura and M. Urago, Identification of embedded interlaminar flaw using inverse analysis, *Int J Fracture* 135 (2005), 153-173.

Collaborations and Technology Transfer

The PI is a key participant in the Center for Thermal Spray technology at Stony Brook University (Director: Prof. Sanjay Sampath) through numerous projects including on-going NSF GOALI project. The thermal spray facility is used to fabricate the initial samples. He also works closely with MesoScribe Technologies, Inc. (www.mesoscribe.com), which manufactures 'Direct Write' sensors of various kinds. Such sensors can be either on-surface or embedded within structures and they are critical components in real-time diagnostics of various materials. The capabilities developed here are ideally suited to process data obtained through these sensors that can be used to measure temperature, strain, moisture, etc. The PI maintains closing working relationship with MesoScribe since the collaborative ARO STTR project, "Integrated Sensing and Modeling for Damage Assessment in Multifunctional Composites" (W911NF-06-C-0180).

Outreach

The PI was the organizer and co-director of DOD sponsored the Long Island Junior Science and Humanities Symposium (JSHS) held at Stony Brook University on March 5, 2011 and March 30, 2012. This year's symposium is scheduled on March 1, 2013 for the 5th time. Nearly 300 high school students submit their research papers and about 80 students are selected to presented their work (www.stonybrook.edu/jshs).

Appendix - Summary of various electrodes

■ Electrode TS Deposition Process

Pre-surface Treatments

1. Grit blast

Surface roughness becomes higher but carbon fibers in the surface layer have to be damaged to exposed fibers.

2. Sand papers

#120: It's too rough because relatively deeper ditches are made on surface.

#240: It is easier to exposed carbon fibers but surface roughness does not become higher.

#400: In order to expose carbon fibers, it is necessary to rub more 10 times in the fiber

direction. After rubbing, surface roughness becomes less than 1 $\,\square m$.

#800: It is difficult to expose carbon fibers.

3. Epoxy remover

Contact resistances between electrode and carbon fibers were not improved. Most likely fibers are recovered with softened epoxy.

Best preparing:

Grid blasting with weak pressure a few second (until the color on the surface is changed) and then rubbing 7 times along the fiber direction of the first layer with sand paper #240.

Thermal Spray

1. Arc spray

Zn can be deposited under any surface conditions.

When Cu is sprayed on Zn electrodes, contact resistances decrease more than 10%.

However, absolute value of contact resistance is quite higher.

2. Plasma spray

Cu electrodes can be made skillfully with spray parameters as shown below:

Spray distance: 150 mm

Ar: 60, H₂: 4

Electrical current: 320 A Power voltage: 60 V

Robot moving speed: 700 mm/s Pass: 7 passes after 1 preheating

Feed: 10 rpm Carrier: 6 lpm

Nozzle diameter: 8 mm Air cooling is from back side

Number of passes:

5 pass: There is no delamination but electrode layer is too thin. 7 pass: Electrodes are finely deposited without delamination.

10 pass: Electrodes are delaminated.

New parameters (Mar 2012) Spray distance: 150 mm

Ar: 47.5, H₂: 3

Electrical current: 400 A Power voltage: 65 V

Robot moving speed: 700 mm/s Pass: 7 passes after 1 preheating

Feed: 10 rpm Carrier: 6 lpm

Nozzle diameter: 8 mm Air cooling is from back side

3. HVOF spray

Surface is burned because of its high-power thermal spray. It is impossible to deposit electrodes on surface of CFRP by HVOF.

Best spray: Plasma spray

■ Electrode Arrangements

Two long electrodes and 3 times 4 circle electrodes (conventional arrangement)
Two long electrodes are used to estimate differences of contact resistance between circle electrodes. The mask size is 12" times 9".

- 1. Circle electrodes for larger size panel Circle electrodes can be deposited on 12" times 18" size panels.
- 2. Long electrodes for larger size panel Long electrodes can be deposited on 12" times 18" size panels.
- 3. Three long electrodes and different size circle electrodes

 Differences of contact resistance between different size electrodes can be measured.

■ Actual Measurement of Resistances between Electrodes

Resistances between electrodes on actual CFRP laminates are measured before and after damaged to obtain resistance changes. And also electrical resistances with the same model are measured after several days to check secular variations of contact resistances between electrode and carbon fibers. In summarized file (measured resistances and surface roughness before thermal spray can be seen. Here each tab name reflects plate name.

4-point probe resistivity measurements

Two electrical wires are deposited on each electrode by soft solder. In general four point probe method, a current source is used to supply current through the outer two probes and the voltage across the inner two probes is measured to determine the resistivity between the two probes. In this study, however, measurement value is not changed even if the two wires on the same electrode are switched.

Artificial Damage

1. Drilling of circular hole.
Any size holes can be made.

2. Grit blasting

Surface damages and holes can be made. It is necessary to use a mask to determine damage size and location accurately. In this experiment, air pressure of grit blast is set at over 60 psi. Here, every 4 or 5 seconds, it is better to check the damage depth which can be measured by counting CFRP layers from the top surface on the verge of the damage. Also, all electric wires have to be secured with tape not to be damaged the other parts.